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Evaluation of Pd-Cr Wires for Strain Gage Application

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Introduction

A newly developed alloy, palladium-13 weight percent chromium (Pd13Cr), was identified by United Technologies Research Center under a NASA contract to be the best material for high temperature strain gage applications [1]. An electrical resistance strain gage that can provide accurate static strain measurement to a temperature higher than that of a commercially available gage is urgently needed in aerospace and aeronautics research. A strain gage made of a 25.4 μm (1 mil) diameter Pd13Cr wire has been recently demonstrated to be usable for static strain measurements to 800 °C [2,3]. This compares to the 400 °C temperature limit of the commercially available strain gages. The performance of this Pd-Cr gage, however, strongly depends on the quality of the Pd13Cr wire. Four batches of Pd-Cr wires purchased from three different manufacturers were therefore evaluated to determine the best source of the wire for strain gage applications. The three suppliers were Precious Metal Institute in China, Sigmund Cohn Co., and G & S Titanium, Inc. in the United States. Two batches of wires obtained from Precious Metal Institute in 1987 and 1992, respectively are referred to herein as China87 and China92 wires. The mechanical, chemical and electrical properties of these wires, both as-received and after high temperature exposures at 800°C for 50 hours were analyzed. The elastic modulus and the failure strength of the wires were evaluated using a tensile test machine equipped with a laser speckle strain measurement system. The chemical and microstructural properties of the wires were inspected using a plasma atomic emission spectrometer and a scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectroscope (EDS). The electrical stability and repeatability of the wires were determined by measuring the electrical resistance of the wires during three thermal cycles to 1000 °C and a ten-hour soak at 1000 °C. As a result of this study, the wire which has the highest strength, the least impurities content, the best oxidation resistance and the best electrical stability will be selected for upcoming strain gage applications.

Mechanical Properties Evaluations

A laser speckle strain measurement technique was used to determine the elastic modulus values of the wires. This laser speckle strain measurement technique initiated by Yamaguchi [4] possesses a small gauge length (0.1 mm) which allows point strain measurements on fibers and wires as small as 21 μm (approximately 1 mil) in diameter [5]. A typical strain resolution of 15 με (micro meter/meter) can be attained using this technique. Details of the laser speckle strain measurement system developed at NASA Lewis can be found in reference 6. In addition to the modulus values obtained from the laser speckle strain

measurement system, the break points for each of the four Pd-Cr wires were determined using the break-point capture feature of a tensile test machine (Instron 4502).

The two ends of the test wires were glued to paper coupons which were then loaded into the grips of the tensile test machine. Each wire was pre-loaded with 100-200 mN of tension, illuminated with 600-700 mW of power from the 514 nm line of the argon laser and pulled at a cross-head velocity of 0.2 mm/minute. The tensile tests were repeated on four samples of each of the four types of wire in the as-received condition. The same sequence of tests was repeated on four samples of each of the four types of wire after heat treatment. The four modulus and break point values obtained for each type of Pd-Cr wire were then averaged and listed in Table I and II for as-received wires and wires after a 50-hour soak at 800 °C, respectively.

Table I: Stiffness and Break-point Strength for As-Received Wire

	Elastic Modulus (GPa)				Break Point (N)			
	China87	China92	Sig-Cohn	G&S	China87	China92	Sig-Cohn	G&S
Sample 1	159	211	160	120	.55	.58	.32	.36
Sample 2	165	133	*	182	.50	.51	.50	.34
Sample 3	135	122	159	162	*	.19	.54	*
Sample 4	188	150	171	175	.42	.56	.21	.32
<avg>	162	168	163	160	.49	.55	.39	.34
<std>	22	37	7	28	.07	.04	.16	.02

Table II: Stiffness and Break-point Strength for Heat-Treated Wires

	Elastic Modulus (GPa)				Break Point (N)			
	China87	China92	Sig-Cohn	G&S	China87	China92	Sig-Cohn	G&S
Sample 1	185	109	156	79	.15	.18	.23	*
Sample 2	82	*	124	88	.15	.19	.22	*
Sample 3	85	128	128	111	.14	.24	.22	.28
Sample	66	113	100	*	.13	.25	.23	*
<avg>	105	117	127	93	.14	.22	.23	.28
<std>	54	10	23	17	.01	.04	.01	*

(*) No data was obtained because the wire was brittle and broke during testing.

There is broad degradation in stiffness and strength of the wires after high temperature exposure. Reduced stiffness is defined as a lower modulus value, and reduced strength is defined as a lower break point value. In comparison, China92 and Sigmund-Cohn (Sig.-Cohn) wires retained more of their original stiffness than the other two wires after the high temperature exposure. The China wires have a higher break strength than the other two wires before annealing, but Sigmund-Cohn edges China92 in break strength after heat treatment. G&S Titanium wires were very brittle after annealing. Consequently, no meaningful average data can be used for comparison.

Chemical Properties Evaluations

A. Chemical Analysis with a Plasma Atomic Emission Spectrometer

An inductively coupled plasma atomic emission vacuum spectrometer was used to determine the composition of the as-received wires. Approximately 0.5 gram of material was used to get a relative accuracy ranging from 1 to 2 percent with multi-element analysis. The composition (in weight percent) of the wires as well as the concentration of major impurities are listed in Table III.

Table III. *Composition of the as-received wires determined by an atomic emission spectrometer*

	Pd (%)	Cr(%)	Si	Fe
China87	87.1	12.6	640 ppm	0.2%
China92	86.9	13.0	20 ppm	30 ppm
Sigmund-Cohn	87.5	11.5	0.2%	0.1%
G&S Titanium	88.6	10.5	0.3%	0.1%

While China92 wire has a Cr concentration of 13% as requested, the other wires are low in Cr concentration. Silicon (Si) and iron (Fe) are detected in all four wires with China92 having the highest purity among these test wires.

B. Structural and Chemical Analysis with a SEM/EDS

A scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectroscope (EDS) was used to study the microstructure of the cross-section as well as the surface of the wires.

I. Cross-section of the wire

The SEM micrographs of cross-section of four as-received wires are shown in figures 1. The diameter of the wires measured from these SEM micrographs are listed in Table IV. Measurements were taken from several areas of each wire with an average value reported in Table IV.

Table IV. Diameter of the as-received wires determined from the SEM micrographs

	China87	China92	Sig-Cohn	G&S
Diameter (μm)	25.0	25.4	26.7	27.1

Note that wires from Sigmund Cohn and G & S Titanium were slightly larger and the China 87 wire was slightly smaller than the 25.4 μm requested size. The dark areas within the wire shown in the SEM micrographs are internal oxides as determined by EDS spectra. In comparison, China92 wire has the least amount of internal oxide among the four wires examined.

The SEM micrographs of the cross-section of the wires after being heat treated at 800°C for 50 hours are shown in Figures 2. The relative X-ray maps of Pd and Cr determined by EDS for these heat-treated wires are shown in Figures 3 and 4. Note from these analyses that Pd diffused to the surface of the wires and formed a Pd rich surface, while O diffused into the surface and formed a Cr rich oxide below the Pd rich zone after the wires were heat treated (oxidized). The diameters of the different layers of the wires are listed in table V.

Table V. Diameter of the heat-treated wires determined from SEM micrographs

(μm)	Overall Diameter	PdCr Diameter	Cr-Oxide Zone Thickness	Pd-rich Zone Thickness
China87	25.2	22.2	0.9	0.6
China92	25.5	23.1	0.8	0.4
Sigmund Cohn	27.0	22.1	0.8-1.6	0.8-1.0
G&S Titanium	27.5	22.4	0.8-1.6	1.3-1.4

In comparison, wires from Sigmund Cohn and G & S Titanium had more Pd migrate to the surface and formed a thicker Cr-rich oxide than the China wires. Sigmund Cohn and G & S Titanium wires therefore degraded more than the China wires after the same heat treatment. Among the two China wires, the quality of the China92 wire was better than the China87 wire. It had less internal oxide and it formed a more uniform and compact oxide than the China87 wire.

II. Surface of the wire

The SEM micrographs of the surfaces of four as-received wires are shown in figure 5. EDS spectra were taken from several areas on the surface of each wire. The average chemical composition (in weight percent) of the wires thus obtained are listed in Table VI.

Table VI. Chemical composition of the wires determined with EDS

wt%	O	Al	Si	Pd	Cr	Others
China87	1.2	0.2	0.4	86.1	12.0	<0.1(S)
China92	1.4	0.1	0.1	86.1	12.3	
Sigmund Cohn	0.9	0.1	0.3	87.8	10.9	<0.1 (Fe)
G & S Titanium	3.1		0.3	87.3	9.3	

In comparison, wires from China have more Cr concentration than the other two sources as was determined by the atomic emission spectrometer. G & S Titanium wire has the lowest Cr concentration and the highest oxygen concentration on the surface which can be related to the different surface structures this wire contains. Silicon was found in all four wires with China92 wire having the lowest Si concentration. S was detected on the surface of China87 wire and Fe on the Sigmund Cohn wire.

The SEM micrographs of the surface of wires after heat treatment are shown in Figure 6. The chemical compositions of the wires (in weight percent) obtained with EDS are listed in the table VII.

Table VII. Chemical composition of the heat-treated wires determined with EDS

wt%	O	Al	Si	Pd	Cr	Others
China87	3.6	0.3		90.0	6.1	
China92	2.1	0.1	0.4	92.4	5.0	
Sigmund Cohn	2.7	1.0	0.2	94.2	1.9	<0.2 (Fe)
G & S Titanium	3.6	0.2	0.4	94.2	1.8	

Note that all the wires have more Pd on the surface after annealing in air at 800°C for 50 hours. Wires from Sigmund Cohn and G&S Titanium had more Pd diffuse to the surface and less Cr remain on the surface after high temperature treatment than the China wires. Greater amounts of impurities diffused to the surfaces of the wires after heat treatment.

Electrical Properties Evaluation

The electrical resistance of these four batches of Pd-Cr wires was measured during three thermal cycles at temperatures ranging from room temperature to 1000 °C with a ten-hour soak at 1000 °C during the second cycle. The resistance of the wires was measured using a four point probe method. The 76 μm Pd-Cr wires which were used as lead wires were spot welded to the 25.4 μm diameter test wires.

The change in resistance versus temperature curves of these four wires are shown in Figure 7, and the resistance drift of the wires during the 10 hour soak are presented in Figure 8. As can be seen, the resistance of the four wires increased linearly with change in temperature up to approximately 815 °C. A break in the resistance versus temperature curves was then observed for all the wires. The break temperature was around 815-850 °C during heating and 760-730 °C during cooling. The magnitude of the break was slightly different among these wires. This break in the resistance vs temperature curve which limits the use of the Pd-Cr wire gage to approximately 800 °C was believed to be due to the reaction of Si and Pd [7]; the higher the concentration of Si impurity in the wire, the higher the magnitude of this break, and the larger the zero shift at room temperature. Note that the resistance of the China wires increased after high temperature exposure while the resistance of the other two wires decreased. The China wires have better electrical resistance stability and repeatability than the other two wires during the same high temperature exposure. The as-received G&S Titanium wire has a higher temperature coefficient of resistance (TCR) than the rest of the wires. This can be related to the lower Cr concentration of the wire.

The average resistance drift at 1000°C for China87, China92, Sigmund Cohn, and G &S Titanium wires are 0.18%/hr, 0.09%/hr, -0.78%/hr and -0.38%/hr, as shown in Fig. 8. Note that the resistance of the China wires increase with time while the resistance of the other two wires decrease with time. China92 wire has the smallest drift and therefore the best stability among these test wires. Sigmund Cohn wire has the highest drift rate or the worst stability among these wires.

Conclusion

The performance of a Pd-Cr wire strain gage, which is useful to 800°C, strongly depends on the quality of the Pd-Cr wire. Of the four wires purchased from three different manufactures examined, China92 wire is the only wire which has the required Cr concentration of 13 weight percent and the required diameter of 25.4 μm. Silicon and Al were the major impurities detected in the four wires. In addition, S was detected in China 87 wire and Fe in Sigmund Cohn wire. China92 wire has the highest break strength and retained more of its stiffness after high temperature exposure. The as-received China92 wire has the least internal oxide, the lowest impurity content and formed the thinnest oxide layer after heat treatment. It therefore has the most stable and repeatable electrical resistance of the four wires. China92 wire will therefore be used to fabricate the PdCr based strain gage for upcoming applications.

References

1. Hulse, C.O., Bailey, R.S. and Lemkey, F.D.,"High Temperature Static Strain Gage Alloy Development Program", NASA Contract Report No. CR-174833, 1985.

2. Lei, J.-F,"A Resistance Strain Gage with Repeatable Apparent Strain to 800°C", J. Experimental Techniques, p.23-27, 1991.

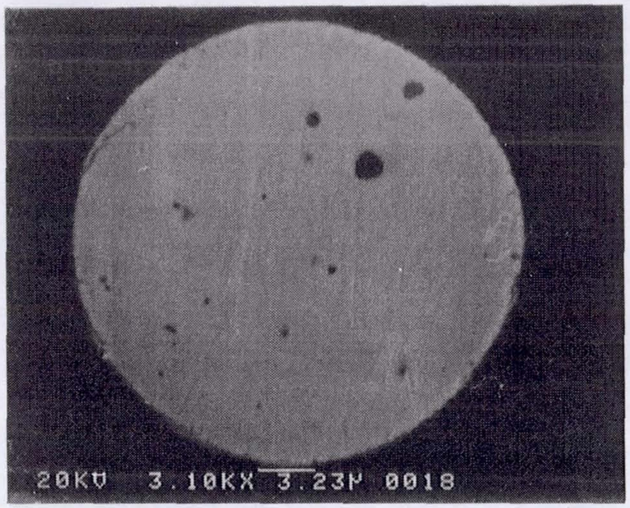
3. Fichtel, E. J. and McDaniel A. D.,"High Temperature Strain Gage Technology for Gas Turbine Engines", NASA CR-191177, 1994.

4. Yamaguchi, I.: "A Laser-Speckle Strain Gauge", J. Phys. E. Sci. Instrum., vol. 14, no. 11, Nov. 1981, pp. 1270-1273.

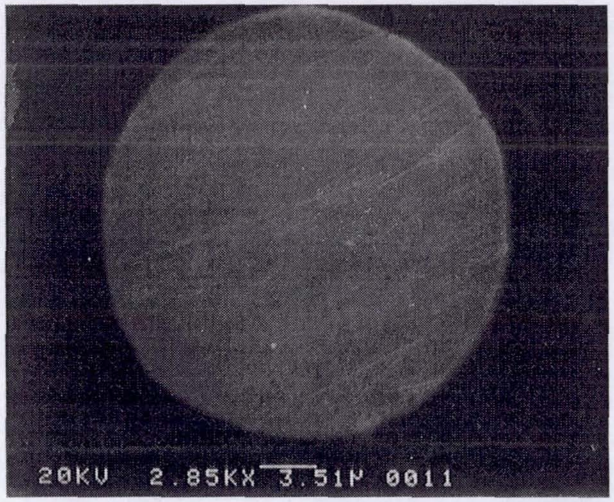
5. Oberle, L.G., Greer, L.C.: "The Laser Speckle Strain Gage: Ambient Temperature Measurements with Small Diameter Fibers", Proceedings of The 5th Annual HITEMP Review 1992, NASA Conference Publication 10104, Vol. 1, pp. 24.1-24.12, 1992.

6. Greer, L.C., Oberle, L.G.: "An Application of the Laser Speckle Shift Measurement Technique for Measuring Strain in Small Diameter Wires and Fibers", Advanced Earth-to-Orbit Propulsion Technology 1992, NASA Conference Publication 3174, Vol. 1, pp. 296-302, 1992.

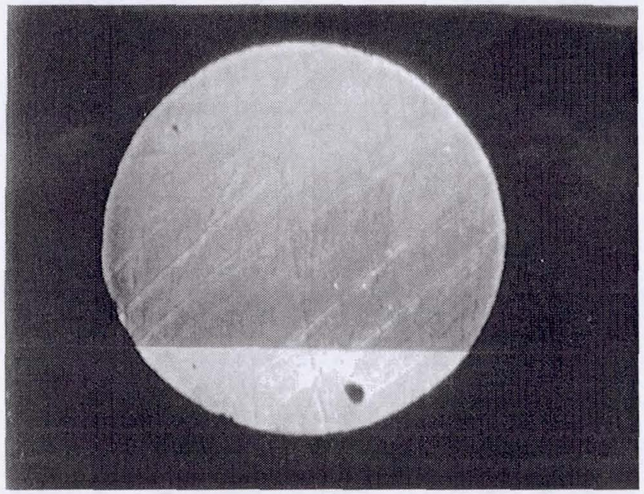
7. Lei, J.-F.,"PdCr Gages Measure Static Strain at High Temperature", Advanced materials & Processed, p.58, May, 1992.



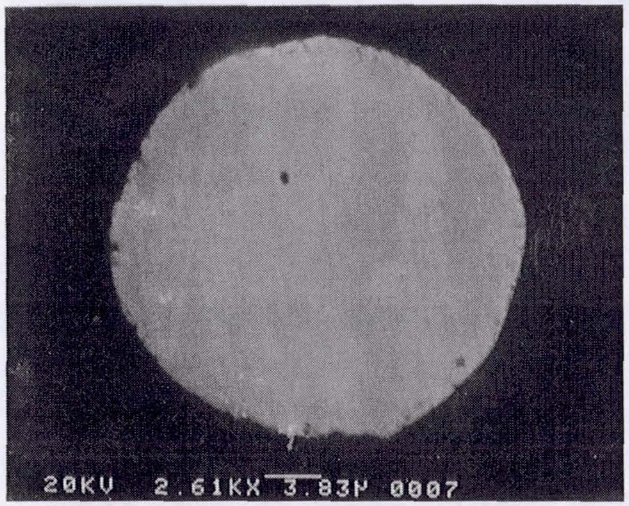
(a) China87 Wire



(b) China92 Wire

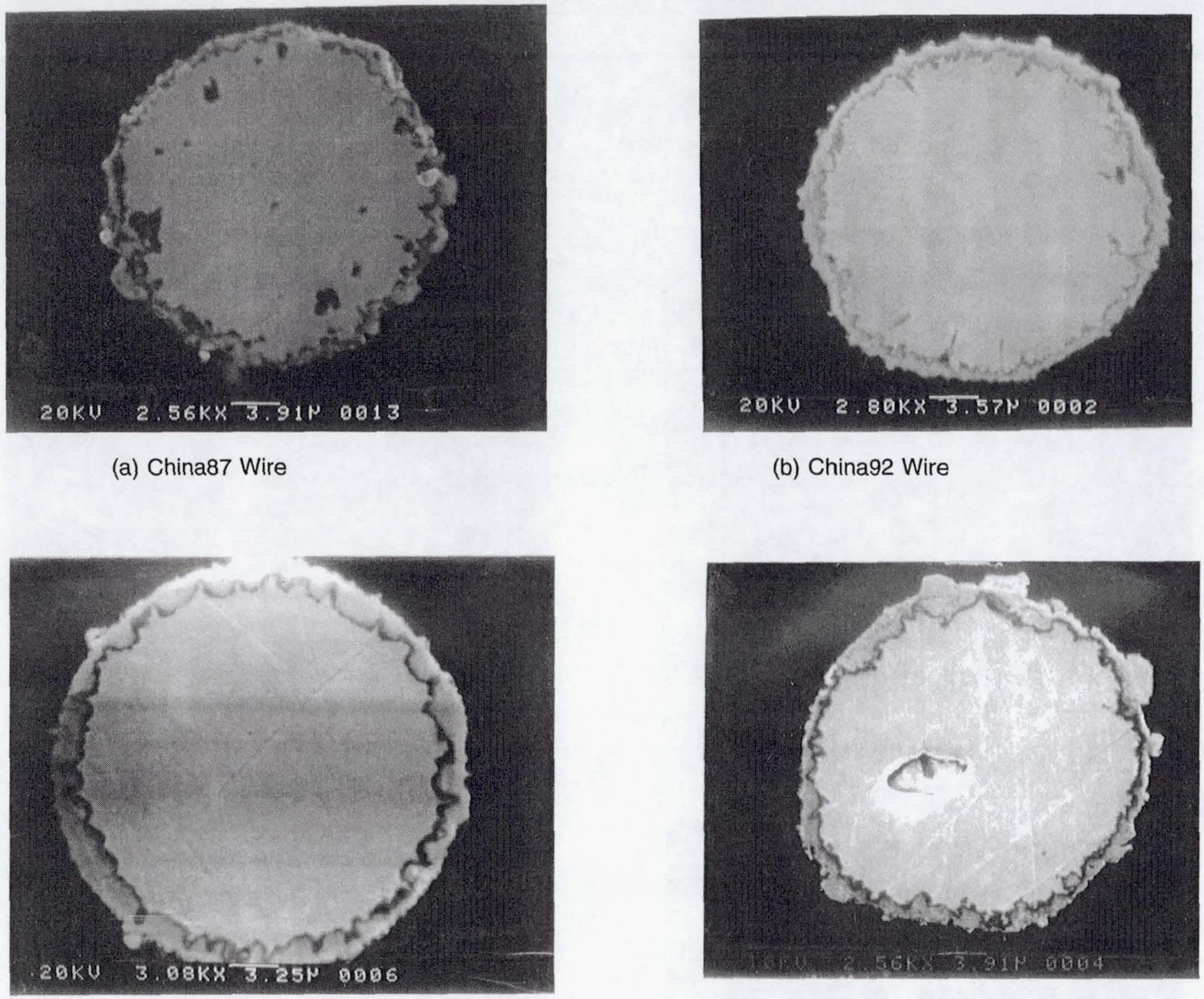


(c) Sigmund Cohn Wire



(d) G & S Titanium Wire

Fig. 1. SEM micrographs of cross-section of four as-received Pd-Cr wires.



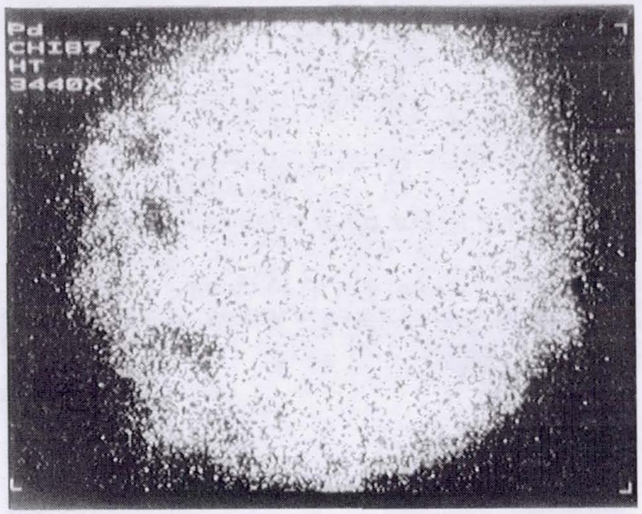
(a) China87 Wire

(b) China92 Wire

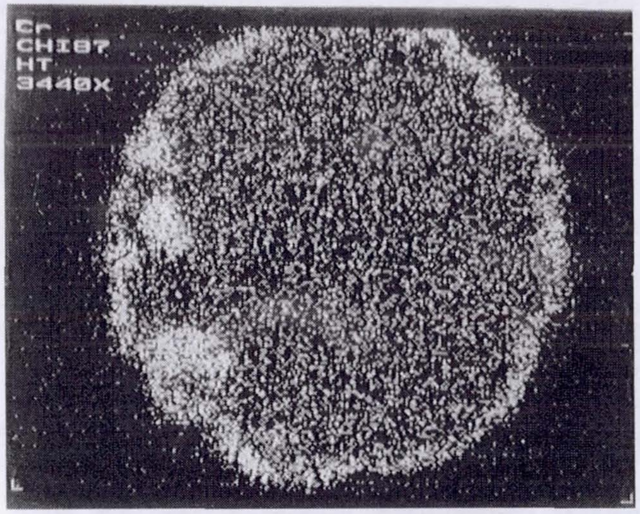
(c) Sigmund Cohn Wire

(d) G & S Titanium Wire

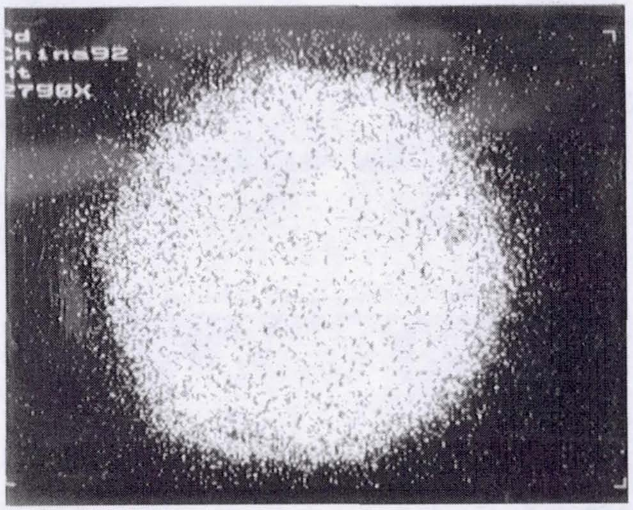
Fig. 2. SEM micrographs of cross-section of four Pd-Cr wires after being heat treated at 800 °C for 50 hours.



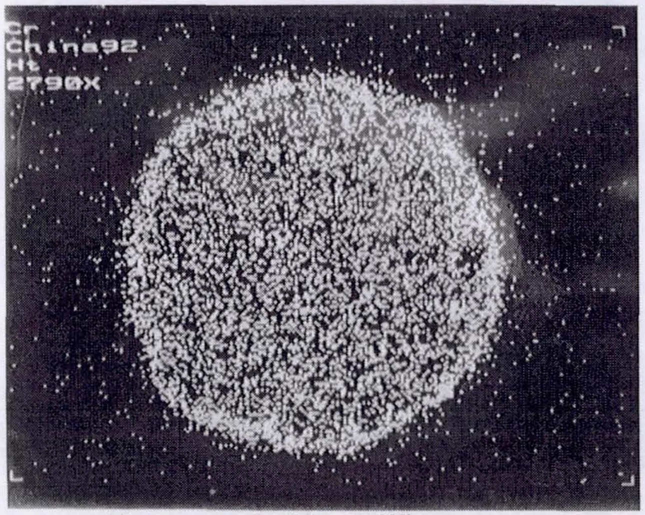
(a) Pd map of China87 Wire



(b) Cr map of China87 Wire

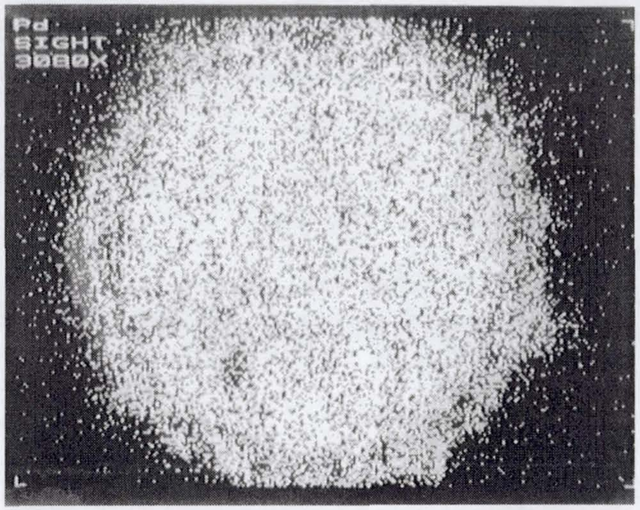


(c) Pd map of China92 wire

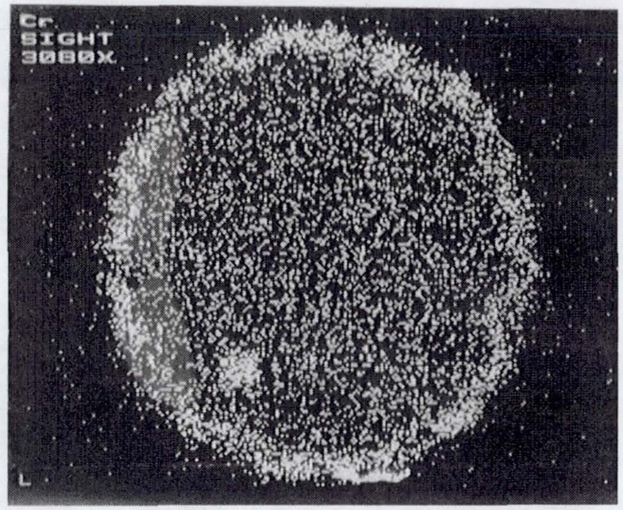


(d) Cr map of China92 Wire

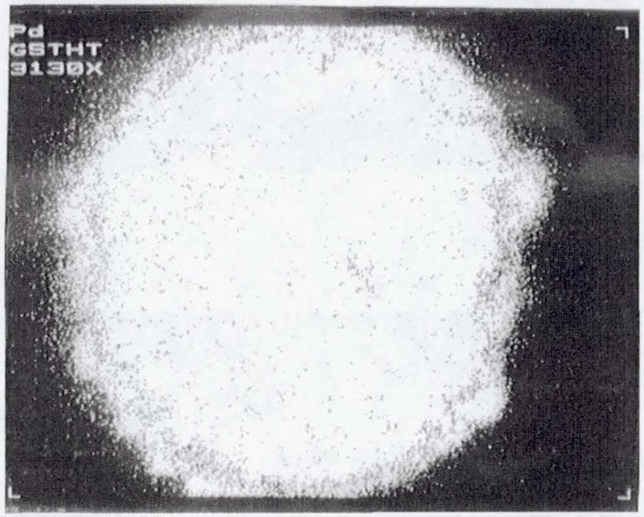
Fig. 3. EDS X-ray maps of Pd and Cr for two heat-treated China wires.



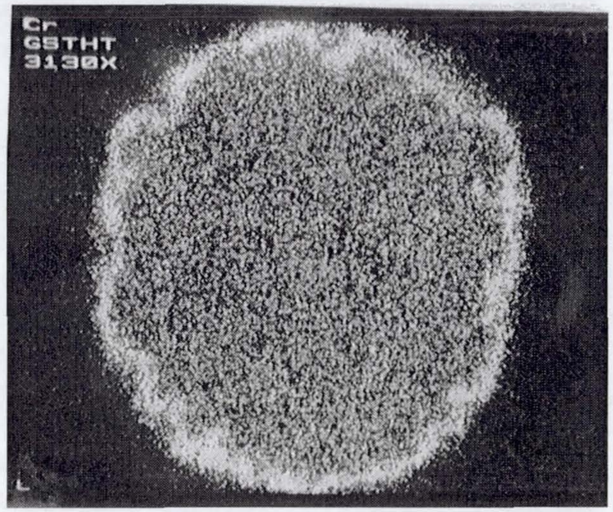
(a) Pd map of Sigmund Cohn Wire



(b) Cr map of Sigmund Cohn Wire

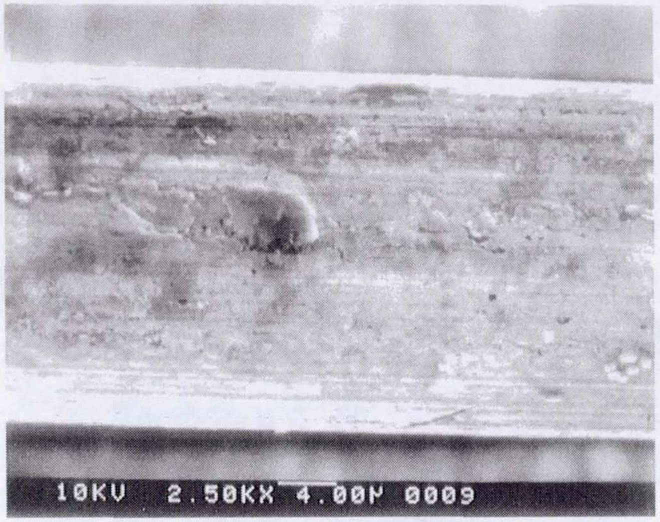


(c) Pd map of G & S Titanium Wire

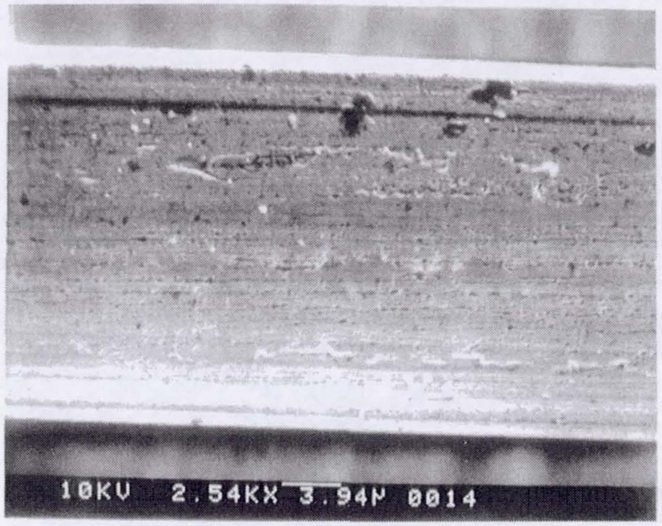


(d) Cr map of G & S Titanium Wire

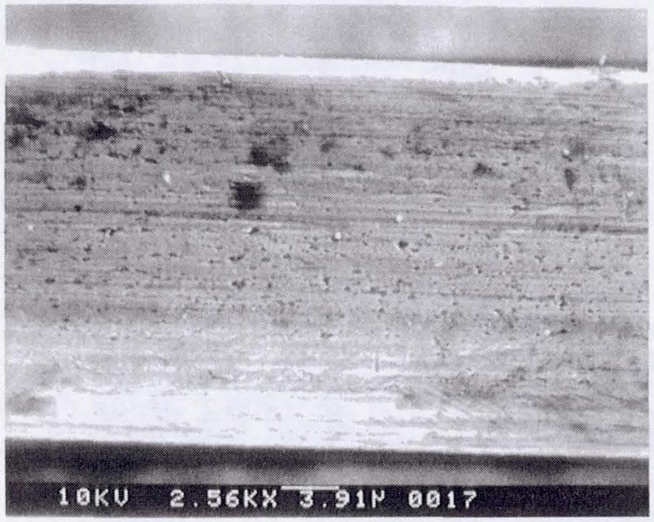
Fig. 4. EDS X-ray maps of Pd and Cr of two Pd-Cr heat-treated wires from Sigmund Cohn and G & S Titanium.



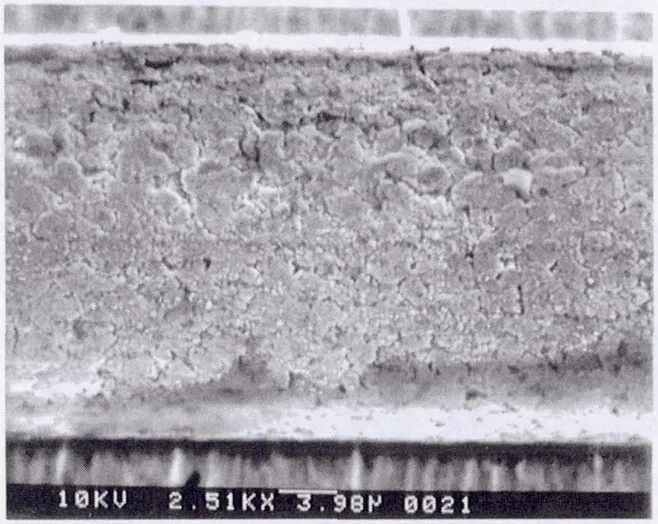
(a) China87 Wire



(b) China92 Wire

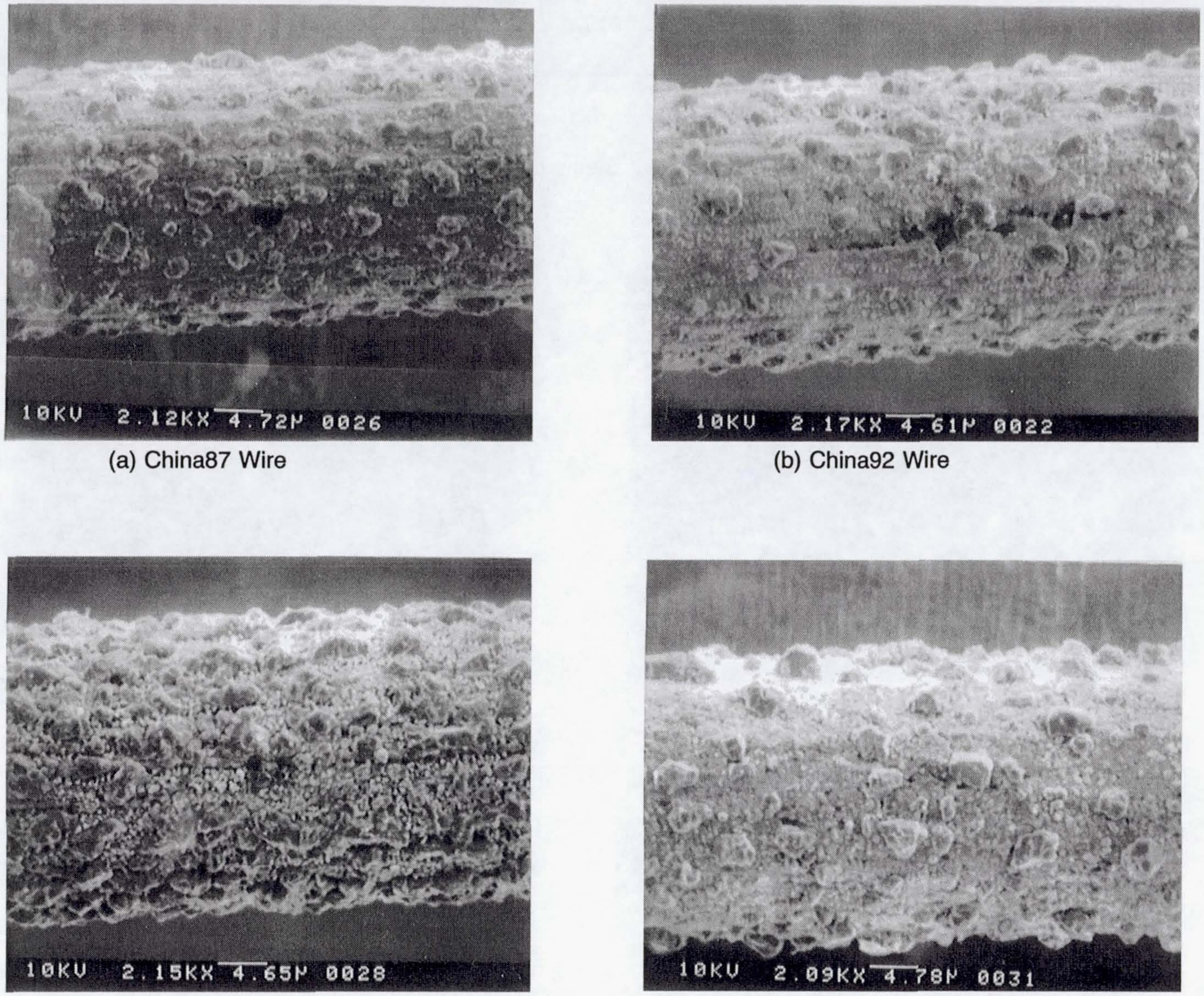


(c) Sigmund Cohn Wire



(d) G & S Titanium Wire

Fig. 5. SEM micrographs of the surfaces of four as-received Pd-Cr wires.



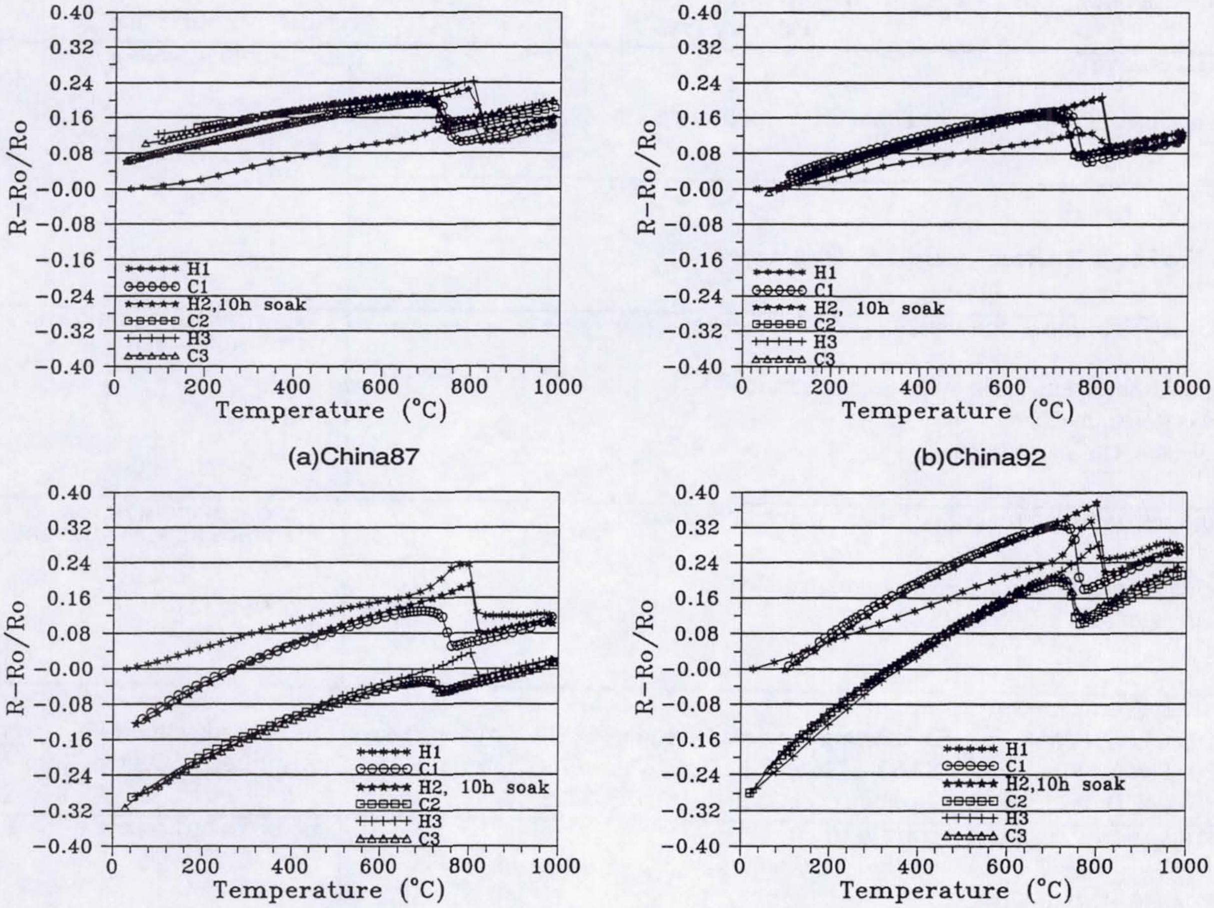
(a) China87 Wire

(b) China92 Wire

(c) Sigmund Cohn Wire

(d) G & S Titanium Wire

Fig. 6. SEM micrographs of the surface of four Pd-Cr wires after being heat treated at 800 °C for 50 hours.



(a)China87 (b)China92

(c)Sigmund Cohn (d)G&S Titanium

Fig. 7. Change in Electrical Resistance vs Temperature of 4 batches of Pd-Cr wires: (a) China87, (b) China92, (c) Sigmund Cohn and (d) G & S Titanium.

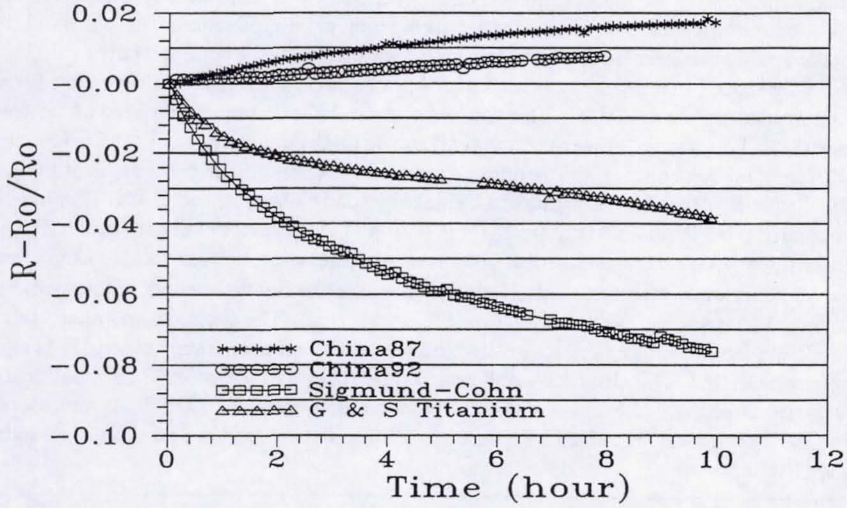


Fig. 8. Change in resistance of the four batches Pd-Cr wires during a 10-hour soak at 1000°C.

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13. ABSTRACT ***(Maximum 200 words)***

A newly developed alloy, palladium-13 weight percent chromium (Pd13Cr), was identified by United Technologies Research Center under a NASA contract to be the best material for high temperature strain gage applications. An electrical resistance strain gage that can provide accurate static strain measurement to a temperature higher than that of a commercially available gage is urgently needed in aerospace and aeronautics research. A strain gage made of a 25.4 μm (1 mil) diameter Pd13Cr wire has been recently demonstrated to be usable for static strain measurements to 800 °C. This compares to the 400 °C temperature limit of the commercially available strain gages. The performance of the Pd-Cr gage, however, strongly depends on the quality of the Pd13Cr wire. Four batches of Pd-Cr wires purchased from three different manufacturers were therefore evaluated to determine the best source of the wire for strain gage applications. The three suppliers were Precious Metal Institute in China, Sigmund Cohn Co., and G & S Titanium, Inc. in the United States. Two batches of wires obtained from Precious Metal Institute in 1987 and 1992, respectively are referred to herein as China87 and China92 wires. The mechanical, chemical and electrical properties of these wires, both as-received and after high temperature exposures at 800 °C for 50 hours were analyzed. The elastic modulus and the failure strength of the wires were evaluated using a tensile test machine equipped with a laser speckle strain measurement system. The chemical and microstructural properties of the wires were inspected using a plasma atomic emission spectrometer and a scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectroscope (EDS). The electrical stability and repeatability of the wires were determined by measuring the electrical resistance of the wires during three thermal cycles to 1000 °C and a ten-hour soak at 1000 °C. As a result of this study, the wire which has the highest strength, the least impurities content, the best oxidation resistance and the best electrical stability will be selected for upcoming strain gage applications.

14. SUBJECT TERMS

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